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A SHOCK TUBE STUDY OF THE DECOMPOSITION KINETICS OF SO_2F_2

Kurt L. Wray and Eugene V. Feldman

AVCO EVERETT RESEARCH LABORATORY

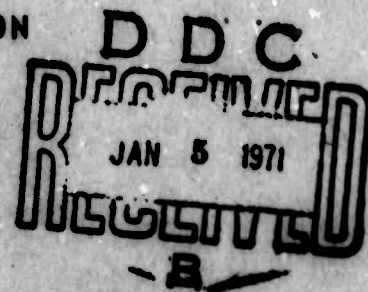### RESEARCH REPORT 348
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by

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AVCO CORPORATION
Everett, Massachusetts

FOREWORD

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Capt. R. W. Padfield, USAF,
Project Officer,
Environmental Technology Branch,
RNSE

ABSTRACT

Recent thermal decomposition studies of SF_6 have led to interest in its principal oxidation product, sulfuryl difluoride. In the present study the thermal stability of SO_2F_2 at high temperatures has been investigated. Highly dilute SO_2F_2-Ar mixtures (~ 0. 1%) were shock heated in a conventional 1. 5" stainless steel shock tube. The SO_2F_2 concentration was monitored as a function of time behind the incident shock wave by its infrared emission at 11.7μ utilizing a liquid helium cooled Cu:Ge detector. The initial pressure in the shock tube was varied from 30 to 600 torr and the temperature range covered was $1900-2300^{\circ}K$. The monitored radiation was shown to be transparent over the range of densities employed. Effective first-order rate constants were evaluated from the logarithmic initial slopes of the radiation decay curves. For the 30-torr data, a unimolecular rate constant fit to the data is $k_{eff} = 2. 1 \times 10^{11}$ exp $(-39,200/T)$ sec^{-1}. The data are analyzed in the light of several modern unimolecular rate theories, yielding ~ 81 kcal as the endothermicity for the reaction

$$SO_2F_2 + Ar \rightarrow SO_2F + F + Ar.$$

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I. INTRODUCTION

Recently there have been a number of experimental studies of the kinetics of the thermal dissociation of SF_6 [1,2]. In the present work we investigated the stability and kinetics of dissociation of sulfuryl difluoride, SO_2F_2, which is (thermodynamically) the principal oxidation product of SF_6. The rate information was obtained in the neighborhood of $2000^\circ K$.

Highly dilute mixtures of SO_2F_2 in Ar were shock-heated to temperatures above which SO_2F_2 is unstable, and its disappearance as a function of time behind the incident shock wave was monitored in emission at 11.7μ.

The emission intensity data, over a wide range of partial pressures of SO_2F_2 showed the gas to be "transparent" in the bandpass employed. The time history data showed the decomposition kinetics to be in the uni-molecular fall off regime over the factor of 20 in pressure covered in the experiment.

The kinetic data have been interpreted employing both the RRK theory[3] and the recently developed complex molecule decomposition theory of Keck and Kalelkar[4]. These analyses indicate an SO_2F-F bond of ~ 81 kcal/mole.

II. EXPERIMENTAL TECHNIQUE

The high purity 1.5 inch stainless steel shock tube used in the present study has been previously described[5]. It is equipped with automatic diaphragm changers and was routinely pumped down to between 10^{-5} and 10^{-6} torr. The IR optical station consisted of gold coated glass mirrors which were used to focus the image of the detector element (1 mm diam. circle) on the center line of the shock tube through an Irtran-4 window. A liquid helium cooled copper-doped germanium detector was used. The filter, placed just in front of the detector, had approximately a bell shaped bandpass, with its peak transmission at 11.75μ and a full width at half height of 0.43μ. The whole system was calibrated with a $400^{\circ}C$ blackbody source.

The SO_2F_2 was obtained from Matheson and is specified 99.5% minimum purity, with air and SO_2 being the main impurities. The pure argon showed no emission in the IR bandpass when shocked by itself. Room temperature IR absorption spectra of a 1% SO_2F_2-99% Ar mixture were run between 2 and 15μ in a 10 cm path length cell at pressures of 10 and 100 torr. Bands ν_1, ν_2, ν_6 and ν_8 appeared as identified by Perkins and Wilson[6]; no other spectral features were found.

Mixtures of SO_2F_2 and Ar were prepared in stainless steel mixing tanks and varied from 0.1% to 1.0% SO_2F_2. Runs were made using initial pressures P_1 from 5 to 600 torr covering the temperature range $\sim$ 1500 - $2300^{\circ}K$. A great deal of this data was used to establish the non-blackness

of the emission lines at temperatures between about 1500 and 1900°K. For the kinetic experiments, where knowledge of the temperature is so important, only the dilute mixtures could be used so as to minimize the temperature drop due to dissociation. The kinetic data were obtained in runs at temperatures between 1900 and 2300°K for the following 4 cases: 0.1% SO_2F_2 at P_1 = 600, 300 and 100 torr and 0.2% SO_2F_2 at P_1 = 30 torr; for these runs conditions were essentially isothermal.

The signal to noise ratio was somewhat of a problem in this experiment; it was typically of order 10. The main sources of noise were the detector and its load resistor and the preamplifier. The time resolution was set by the shock transit time across the detector image and the electronic rise time of the detector circuit, these two being of the same order of magnitude. The net effective risetime in the kinetic experiment was 3.7 and 5.5μ sec (10% - 90% signal risetime), the faster one being used at the higher temperatures to allow resolution of the faster chemistry.

III. EXPERIMENTAL RESULTS

Two typical oscillograph traces are shown in Fig. 1 representing (a) the low temperature ($1690^{\circ}K$) and (b) the high temperature ($2080^{\circ}K$) data. At low temperatures ($1500 - 1900^{\circ}K$), the IR signal is seen to be essentially a step function, the rise time of which is instrumental. At the temperature of the run shown in Fig. 1a, the SO_2F_2 is either stable or dissociates so slowly that the IR emission stays constant following vibrational excitation. In Fig. 1b, the temperature is sufficiently high so that the SO_2F_2 decomposes subsequent to vibrational relaxation; hence, the IR signal decays as a function of time.

These considerations are illustrated in Fig. 2 where the equilibrium concentrations have been plotted as a function of temperature for the case 0.1 torr initial partial pressure of SO_2F_2, e.g., 0.1% SO_2F_2-99.9% Ar, P_1 = 100 torr shock conditions. Two cases are shown: (1) the enthalpy of formation of SO_2F was assumed to be ΔH_f ($298^{\circ}K$) = -143 kcal/mole (SO_2F-F bond = 81 kcal) and (2) ΔH_f ($298^{\circ}K$) = -129 kcal/mole (SO_2F-F bond = 95 kcal). The later case corresponds very nearly to omitting SO_2F completely as an allowed species in so far as the stability of SO_2F_2 at high temperature is concerned. To keep the figure relatively simple, the F-atom concentration was not plotted, nor were SO, O_2, O and S at the higher temperatures where the SO_2 is dissociating. These equilibrium calculations were carried out on a computer using as input the thermodynamic functions given by Wilkins[7] for SF_6 and its decomposition and oxidation products; however, SO_2F was not considered in

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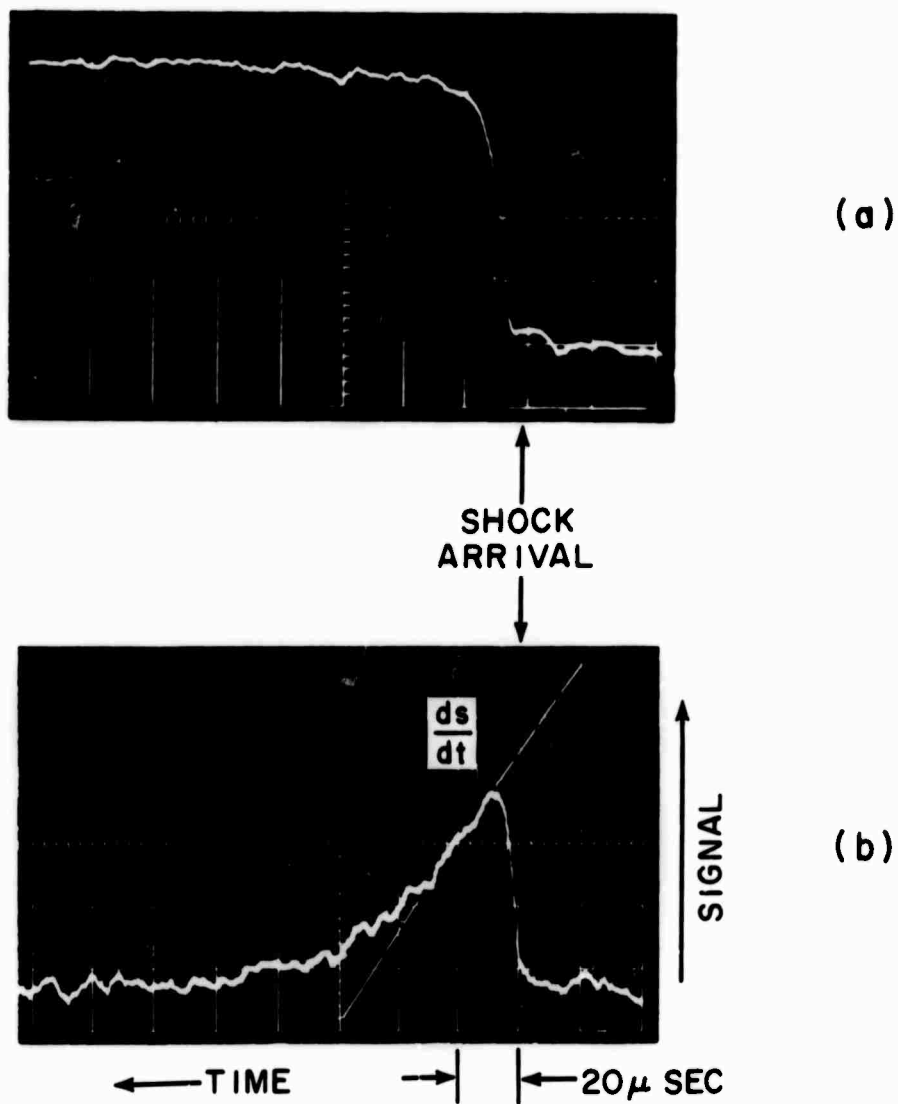
Fig. 1 Typical oscillograph traces showing: (a) No dissociation; $U_s = 1.26$ mm/μ sec, $P_1 = 30$ torr, $SO_2F_2 = 1.0\%$, $T = 1690°K$, and (b) Dissociation; $U_s = 1.42$ mm/μ sec, $P_1 = 300$ torr, $SO_2F_2 = 0.1\%$, $T = 2080°K$.

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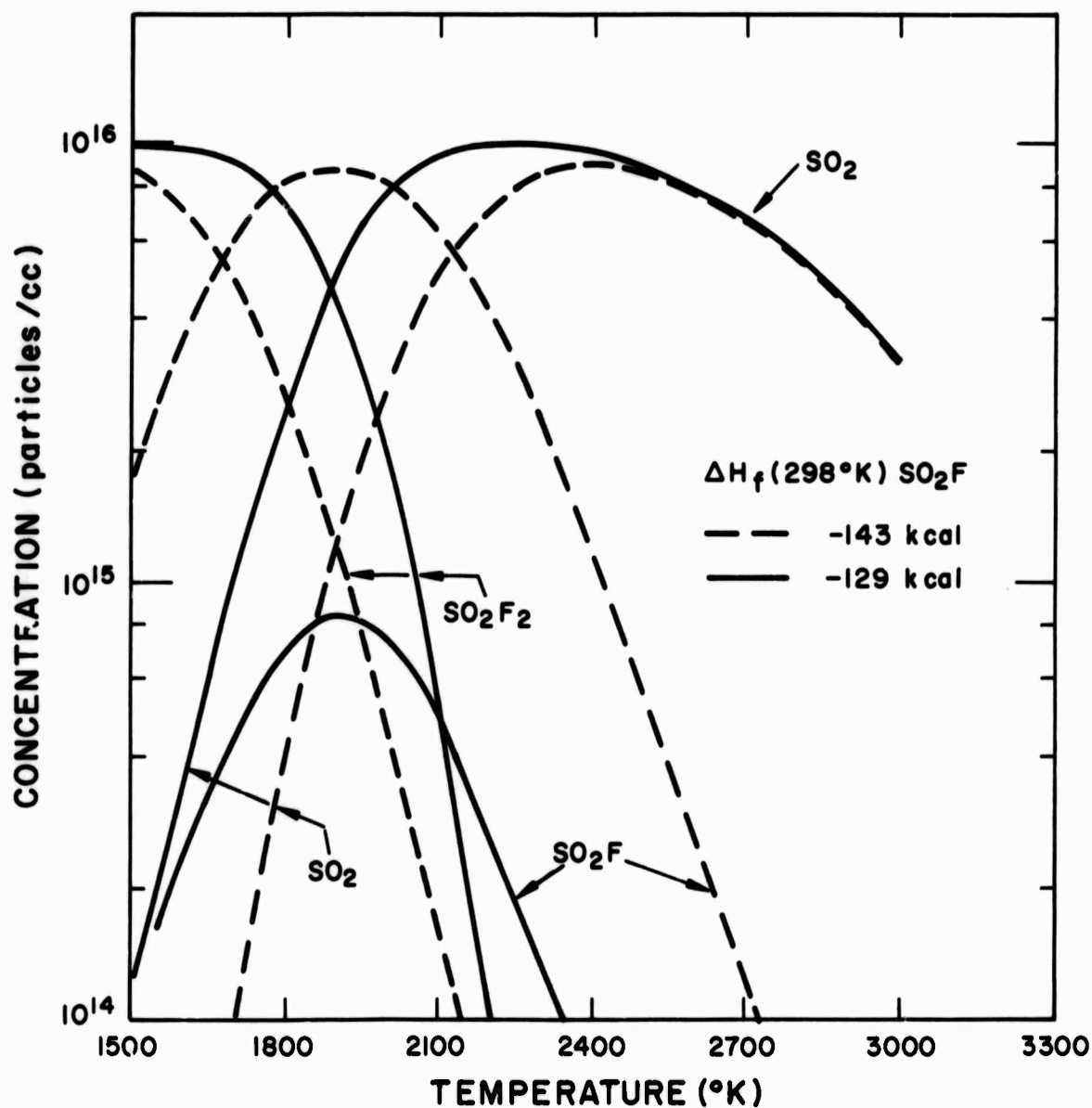
Fig. 2 Equilibrium concentrations of SO_2F_2, SO_2F and SO_2 as a function of temperature for initial shock conditions $P_1 = 100$ torr, $SO_2F_2 = 0.1\%$. Two cases are shown, i.e., SO_2F heats of formation of -143 and -129 kcal/mole, corresponding to SO_2F-F bond strengths of 81 and 95 kcal, respectively.

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Ref. 7. As shall be seen below, the present experiments indicate an SO_2F-F bond strength of $\sim$ 81 kcal, and hence at intermediate temperatures it becomes a major specie. The thermodynamic properties of the SO_2F were estimated for us by Wilkins[8].

It would have been highly desirable to measure the SO_2F-F bond strength directly in the present work by monitoring the equilibrium concentration of SO_2F_2 in the sensitive temperature range between 1600 and 2000°K. However, shock attenuation and boundary layer growth effects make the temperature uncertain by up to several hundred degrees by the time equilibrium is established ($\sim$ 100 μsec in the present experiment). Inspection of Fig. 2 shows that this magnitude of temperature uncertainty does not allow determination of the bond strength to the desired accuracy by this method.

With the present apparatus, meaningful kinetic data could not be obtained above about 2300°K due to insufficient resolution. Below about 1900°K, both the small amount of decomposition and the slow rate of decomposition prohibited kinetic measurements as the effects of shock attenuation were noted to become dominant under these conditions.

The data obtained in the temperature range 1500 - 1900°K were used to demonstrate the transparency of the emitting gas. For a thin gas, the intensity in a band is given by

$$ I = a N_\lambda \, l \, \lambda^2 \, \left\{ SO_2F_2 \right\} \qquad watts/cm^2\text{-ster} , \qquad (1) $$

where a is the integrated band intensity in $cm^{-2} atm^{-1}$, N_λ is the value of the blackbody function at the radiating gas temperature in $watts/cm^2$-

ster-cm, l is the optical path length in cm, λ is the wavelength in cm and $\left\{SO_2F_2\right\}$ is the concentration of SO_2F_2 in amagats:

$$\left\{SO_2F_2\right\} = \frac{P_1}{760} \frac{\rho_2}{\rho_1} \frac{273}{T_1} f . \tag{2}$$

where P_1 is the initial shock tube pressure in torr, ρ_2/ρ_1 is the density ratio across the shock, T_1 is the initial temperature of the test gas and f is the fraction of SO_2F_2 in the test gas mixture. Equations (1) and (2) indicate that for a thin gas

$$\frac{1}{P_1} \propto f . \tag{3}$$

The plateau values of the IR signals, normalized by the pressure, have been plotted against the percent of SO_2F_2 in Fig. 3. Each one of these data points is an average of from 3 - 10 separate runs covering a temperature range of 1500 - 1900°K. Within the scatter in the data, the temperature dependence over this range was negligible. The data fall on a line of unit slope on the log-log plot indicating agreement with Eq. (3) and, hence, that the emission lines are not black.

It was important to show that under the experimental conditions, Eq. (3) is followed. This unambiguously shows that the radiation signal is proportional to the SO_2F_2 concentration, thus allowing interpretation of the kinetic data. Before leaving the data shown in Fig. 3, however, it is of some value to use it to obtain an estimate of the integrated band intensity a. We note that
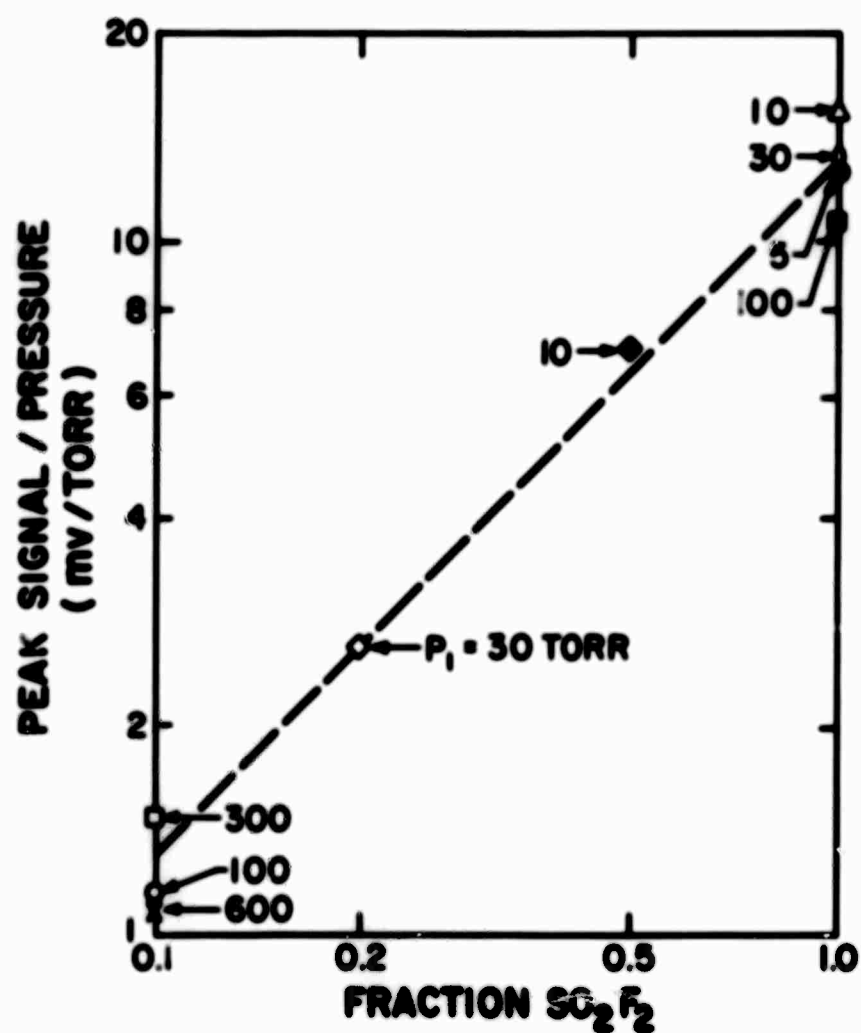
Fig. 3 Peak 11.7μ radiation signals, normalized with the initial shock tube pressure plotted vs. percent of SO_2F_2 in the gas mixture. Each data point is the average of many runs; $1500 \leq T \leq 1900°K$.

$$a = \frac{1}{P_1} \, 760/N_\lambda \; l \; \lambda^2 \frac{\rho_2}{\rho_1} \frac{273}{T_1} \, l \quad . \tag{4}$$

and that $1/P_i$ is obtainable from Fig. 3 by use of the blackbody calibration factor J and the bandpass of the filter $\Delta\lambda = 4.5 \times 10^{-5}$ cm:

$$\frac{i}{P_i} = \frac{S_R \, J \, \Delta\lambda}{P_1} \quad \text{and} \quad J = \frac{N_{\lambda (B)}}{S_B} \quad . \tag{5}$$

where S_R and S_B are the detector signals for a run and calibration, respectively, and $N_{\lambda (B)}$ is the value of the blackbody function at the calibration source temperature. From Eqs. (4) and (5) and the data of Fig. 3 we find $a \approx 500 \text{ cm}^{-2} \text{ atm}^{-1}$.

It should be pointed out that the above value of a is, at best, an approximate one. At room temperature[6] the $\nu_2 (A_1)$ band at 11.8μ (848 cm^{-1}) is just about encompassed by the bandpass filter used in the present experiment, and the $\nu_8 (B_2)$ band at 11.3μ (885 cm^{-1}) is just about excluded. However, at elevated temperatures, both bands must spread in wavelength so that the experimental bandpass must exclude some of the ν_2 and include some of the ν_8 radiation.

The high temperature data were used to obtain the dissociation rate constant. For the reaction

$$SO_2 F_2 \rightarrow \text{products} \tag{R1}$$

an experimental (effective) unimolecular rate constant k_{eff} is defined by:

$$\frac{i}{[SO_2F_2]} \frac{d[SO_2F_2]}{dt_p} = k_{eff} \quad . \tag{6}$$

where $[SO_2F_2]$ is the concentration of SO_2F_2 in particles/cc and t_p is the particle time in sec. Noting that $S \propto [SO_2F_2]$, where S is the detector output signal, and that $t_p = \dfrac{\rho_2}{\rho_1} t_L$ where t_L is time in the laboratory coordinate system, we have

$$\frac{1}{S}\frac{dS}{dt_L} \cdot \frac{\rho_1}{\rho_2} = \frac{1}{S}\frac{dS}{dt_P} = k_{eff} \cdot \qquad (7)$$

For the kinetic runs $\rho_2/\rho_1 \cong 3.5$ independent of mixture or shock speed. Such a logarithmic derivative can be taken any place in the time history provided that the decay is truly exponential. Non-exponential decay is observed at long times when attenuation effects become non-negligible, and at low temperatures where the backward reaction becomes important. dS/dt_L is indicated on the oscilloscope trace shown in Fig. 1b.

The kinetic data are presented in Fig. 4 as an Arrhenius plot of k_{eff} vs. reciprocal temperature. The four straight lines are least square fits to the four sets of data assuming an Arrhenius type temperature dependence. For the 30 torr case

$$k_{eff} = 2.1 \times 10^{11} \exp(-39,200/T) \text{ sec}^{-1} \quad , \qquad (8)$$

which corresponds to an activation energy of $E = 78$ kcal/mole.

The density corresponding to the data for the extreme P_1 cases are indicated in the figure as ρ_2/ρ_0, where ρ_0 is standard density. It is of some interest to compare the rate of decomposition of SO_2F_2 with its parent compound, SF_6. This is done in Fig. 4 at 2000°K where the range of k_{eff} for SF_6 decomposition is shown for the same density range as encompassed by the present data. This information was obtained by cross plotting the data of Bott and

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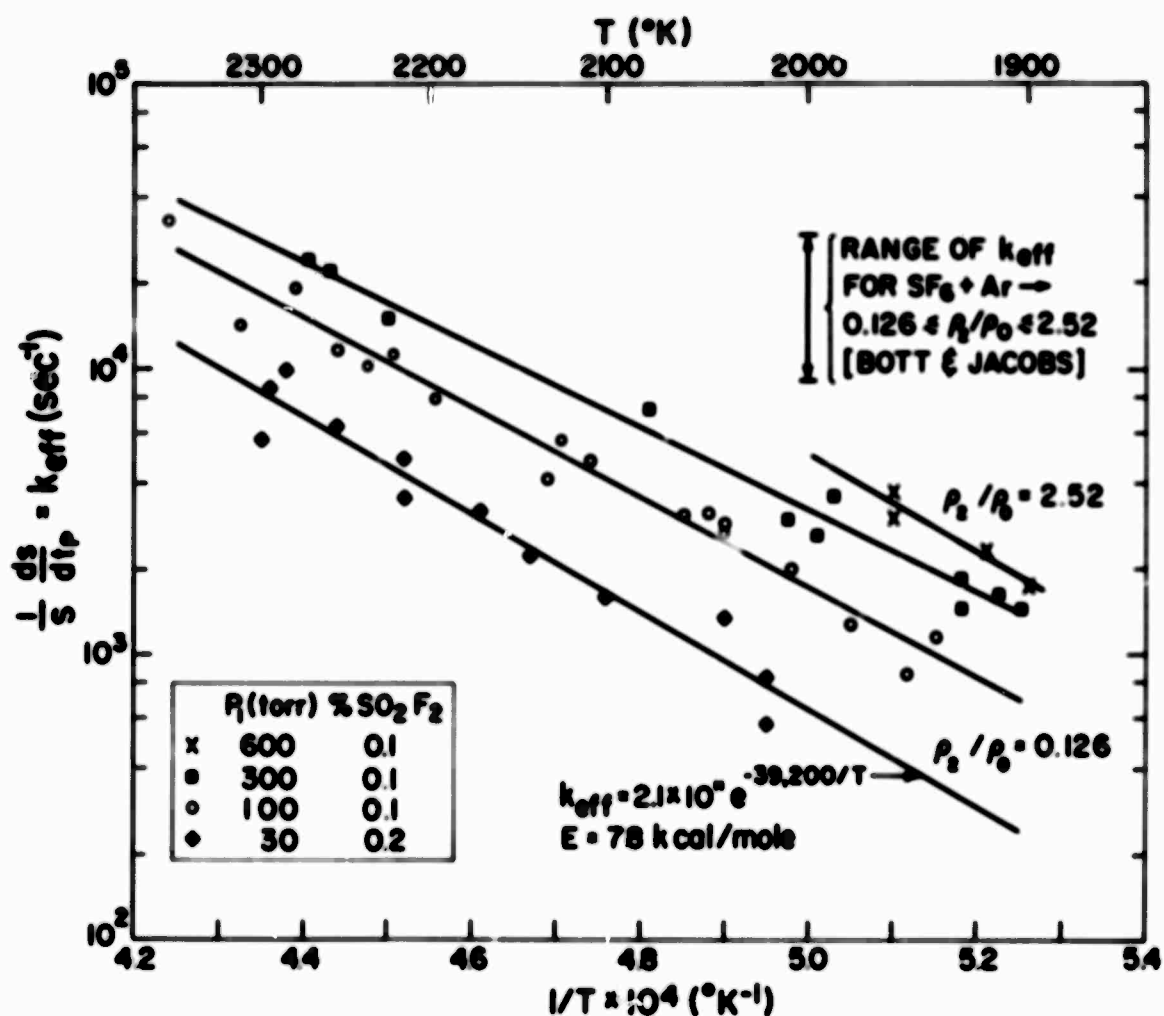
Fig. 4 Effective rate constant for SO_2F_2 decomposition by Ar
determined from the initial slopes plotted vs. reciprocal
temperature. The straight lines are least square fits to
the data taken at the four initial pressures of $P_1 = 30$, 100
300 and 600 torr. Shown for comparison is the rate constant
for SF_6 decomposition over the same density range.

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Jacobs.[1] It is seen that SF_6 decomposes about 10 times faster than does its oxidation produc:, SO_2F_2.

The limits of P_1 and T were dictated by the experiment: low partial pressures of SO_2F_2 were required to maintain isothermal conditions; at low temperatures the slow rate and stability of SO_2F_2 led to flat signals; at high temperatures rise time limitations prevailed; at low pressures the data were noise limited, and at high pressures severe shock attenuation became dominant.

IV. COMPARISON WITH THEORY

The kinetic data have been analyzed within the framework of two complex molecule dissociation theories, i.e., those of Rice, Ramsperger and Kassel (RRK)[3] and Keck and Kalelkar[4]. The well known RRK theory has in it a number of adjustable parameters. Figure 5 shows the sensitivity to these parameters when the experimental data is compared to the theory. In this figure k_d/k_∞ is plotted against $\log_{10} (A/\lambda\omega)$, where k_d is the theoretical dissociation rate constant which is normalized by k_∞ - the limiting high pressure rate constant, A is the frequency factor, and λ is the collision efficiency. The collision frequency ω is given by

$$\omega = \pi\sigma^2 \left(\frac{8}{\pi\mu kT}\right)^{1/2} P \quad , \tag{9}$$

where σ is the mean collision diameter, μ is the reduced mass for the collision pair, and P is the total pressure of the system.

The high pressure limit to the rate constant is given by

$$k_\infty = A \exp(-E/kT) \quad , \tag{10}$$

where E is the energy of the bond which is broken when the reactant molecule dissociates.

The rate constant is given by

$$k_d/k_\infty = I(S, E/kT, \log A/\lambda\omega) \quad , \tag{11}$$

where I is the Kassel integral, and S is the number of classical harmonic oscillators that couple to the dissociating bond, this usually being about 1/2 of the total number of vibrational modes. Tables of this function have been prepared by G. Emanuel.[9]

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Fig. 5 Sensitivity of RRK theoretical rate constant to the theory
parameters and comparison with data at 2000 and 2200°K.
The parameters S and A were varied for assumed bond
strengths of 81 and 95 kcal.

Hence, the parameters which are needed are σ, λ, A, S and E. The molecular diameter of SO_2F_2 is not known; a value of $\sigma = 4$ Å for the Ar - SO_2F_2 collision pair was chosen and cannot be in error by a significant amount. Following the work of Bott & Jacobs[1], λ was set equal to 1/4. A, S and E were adjusted to obtain the best fits with the data. It was assumed that the reaction mechanism was

$$SO_2F_2 + Ar \rightarrow SO_2F + F + Ar \quad , \qquad R2$$
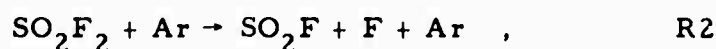
as this clearly has the lowest activation energy of the several conceivable SO_2F_2 decomposition paths. Two values of E were considered, 81 and 95 kcal. The latter was chosen, rather arbitrarily, based on the average S-F bond energy of 86 kcal in SO_2F_2. The value of 81 kcal was estimated by Wilkins[8].

As is seen in Fig. 5, F = 95 requires S = 7 or larger at T = $2000^{\circ}K$; the best, but not very satisfactory, fit being made with $A = 3 \times 10^{14}$ sec^{-1}. The E = 81 case can be only poorly fit as S = 5 with $A = 4 \times 10^{12}$ but is nicely fit at S = 4 with $A \approx 1 \times 10^{13}$. Similarly, a comparison of theory and data at $2200^{\circ}K$ shows that E = 81, S = 4 and $A = 1 \times 10^{13}$ is a good fit (see Fig. 5).

In Fig. 6, all the data is compared to the RRK theory in the k_d, 1/T plane for the best results for the cases E = 95 and 81 as shown in Fig. 5. Here it is seen that the case E = 81, S = 4, $A = 1 \times 10^{13}$ fits the data adequately, while the slope of the E = 95 case is too steep. A frequency factor of 1×10^{13} sec^{-1} is quite typical for reactions of the type studied in the present work and S = 4 is "reasonable" for the SO_2F_2, which has 9 vibrational degrees of freedom.

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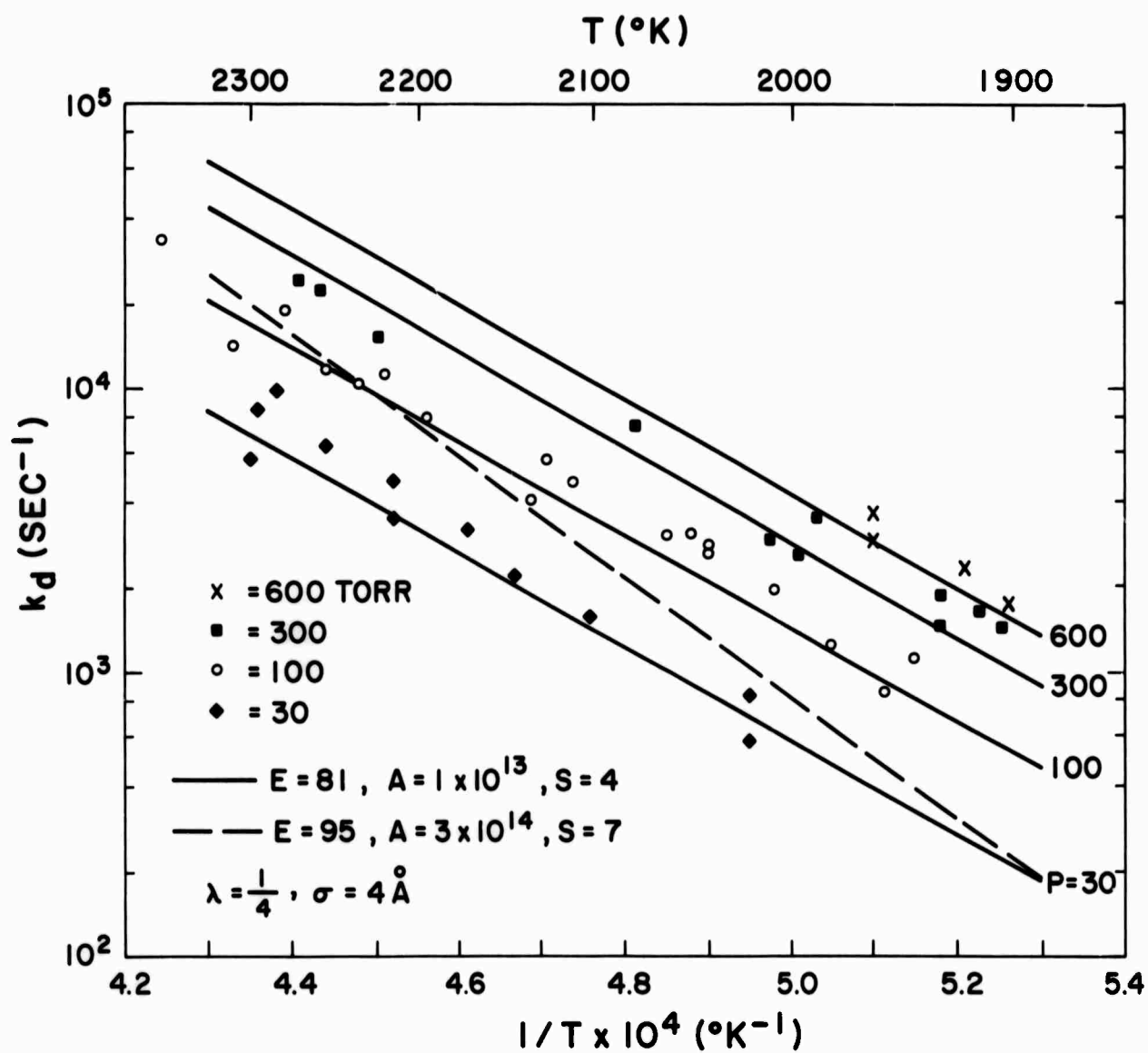
Fig. 6 Comparison of experimental and RRK theoretical rate constants as a function of reciprocal temperature for the best fit cases of Fig. 5 for assumed bond strengths of 81 and 95 kcal.

The theory of Keck & Kalelkar[4] is similar to that of RRKM[10] in its basic assumptions but it differs in detail and the results are in a simple form which facilitates computations. A successful correlation of k_d for 21 molecules having 3-6 atoms has been obtained with this theory[4]. The theory has three adjustable parameters, E, σ_{1A} and σ_{23}; E has the same interpretation here as in the RRK theory, σ_{1A} is the effective energy transfer cross section and σ_{23} is the effective spontaneous decay cross section. These cross sections, within the framework of the theory, are not completely adjustable but $\sigma_{1A} \approx 3 \, Å^2$ and, for the present reaction where one bond is broken and an atom is one of the products, $\sigma_{23} \approx 1 \, Å^2$.

A comparison between the data and the Keck theory at 2000^oK is shown in Fig. 7, where $\log k_d/\sigma_{23}$ is plotted against $\log \frac{\rho_2}{\rho_0} \frac{\sigma_{1A}}{\sigma_{23}}$. It is seen that for the case $\sigma_{1A} = 3 \, Å$, $\sigma_{23} = 1 \, Å$, as called for by the theory, the comparison between theory and experiment is very poor, and E > 95 kcal is required. Better fits can be made at E = 81, but $\sigma_{1A} = \sigma_{23} = 0.04$ for this case, which is not allowed by the model. In the k_d vs. 1/T plane, the Keck theory does not fit the data well either.
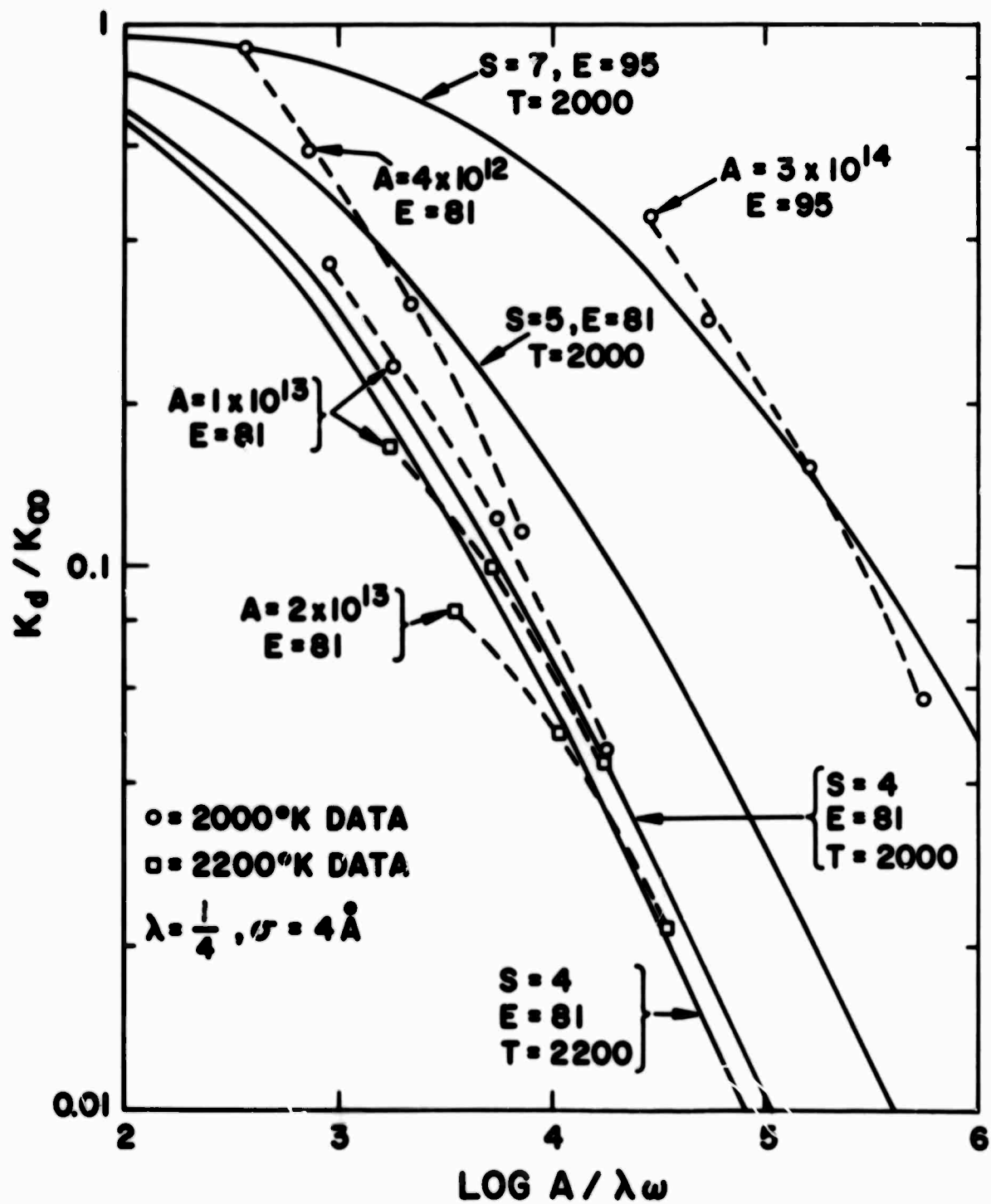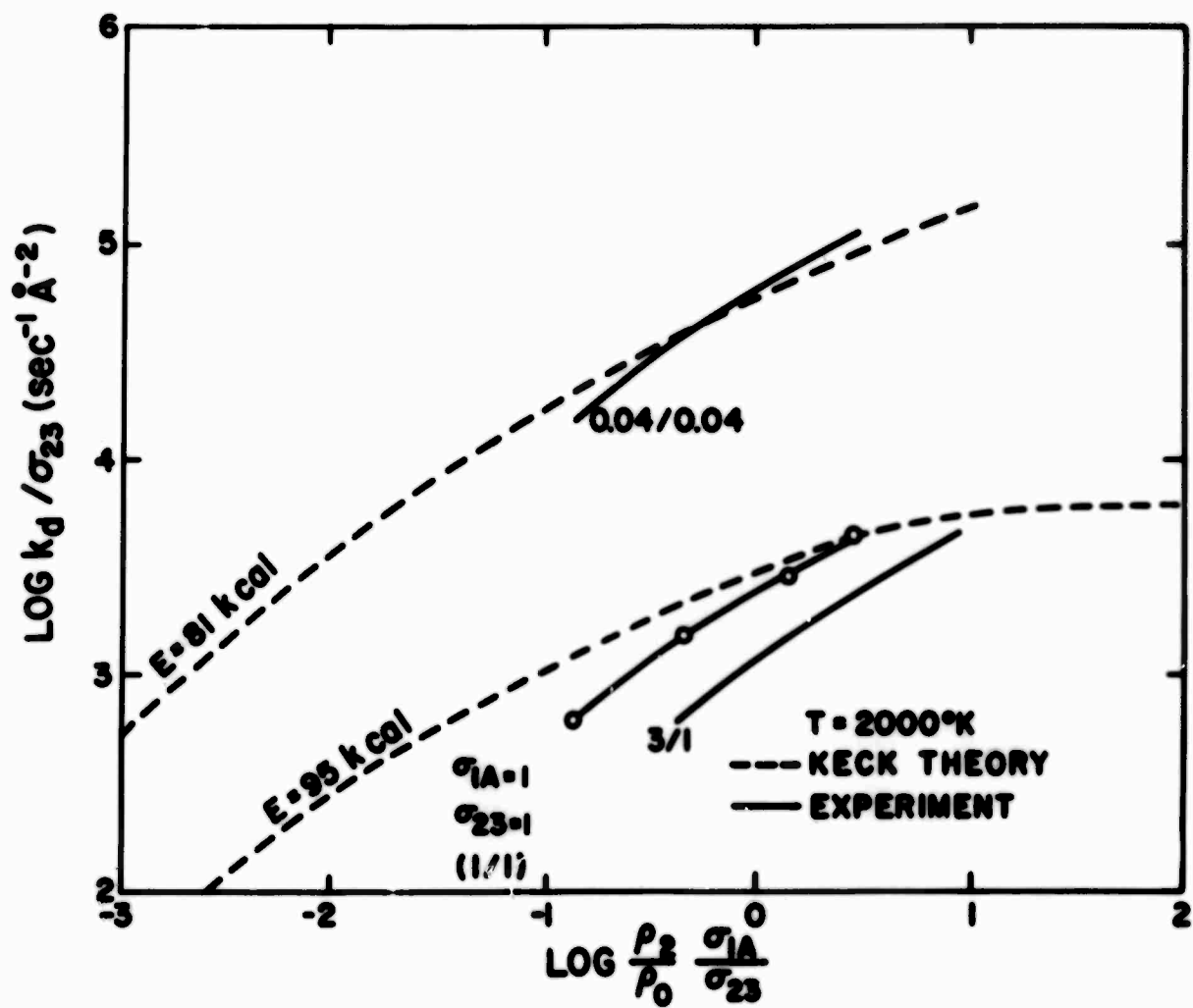
Fig. 7 Sensitivity of Keck theoretical rate constant to the theory
parameters and comparison with data at 2000°K. The
parameters σ_{1A} and σ_{23} were varied for assumed bond
strengths of 81 and 95 kcal.

V. ACKNOWLEDGMENT

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13 ABSTRACT

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	KEY WORDS	LINK A		LINK B		LINK C	
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2.	Thermal Decomposition						
3.	Unimolecular Reaction Theory						
4.	Shock Tube						
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